

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : **09-323204**

(43)Date of publication of application : **16.12.1997**

(51)Int.Cl.

**B23B 27/14**

**C22C 29/00**

**C23C 14/06**

(21)Application number : **08-166809**

(71)Applicant : **HITACHI TOOL ENG LTD**

(22)Date of filing : **05.06.1996**

(72)Inventor : **SHIMA NOBUHIKO**

### (54) MULTILAYER COATED HARD TOOL

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a cutting tool further improved in oxidation resistance by reducing residual compressive stress, realizing the thickening of a film and adding a prescribed third component to TiAl, in a TiAl film.

SOLUTION: A tool is coated with at least two or more layers of a layer composed of Ti nitride or carbonitride and a layer composed of the nitride or the carbonitride of Ti, Al and a third component. The value of the I (200)/I (111) of the Ti nitride or carbonitride layer is made 1 or below and the value of the I (200)/I (111) of the nitride or carbonitride layer of Ti, Al and the third component is made 1 or more. The third component is composed of a kind or two or more kinds of components selected among Zr, Hf, ~~Cr~~, W, Y, Si, Ce and Nb.

### LEGAL STATUS

[Date of request for examination] 31.07.1997

[Date of sending the examiner's decision of rejection] 14.08.2001

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection] 2001-16312

[Date of requesting appeal against examiner's decision of rejection] 13.09.2001

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

## \* NOTICES \*

**Japan Patent Office is not responsible for any damages caused by the use of this translation.**

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

DETAILED DESCRIPTION

---

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the covering hard tool which has the outstanding abrasion resistance.

[0002]

[Description of the Prior Art] aluminum is made to contain to common TiN or TiCN coating conventionally in recent years, the research which raises abrasion resistance and oxidation resistance is made, and various examples which accept the addition effect of aluminum also exist so that it may be represented by JP,4-53642,B and JP,5-67705,B. Moreover, an artificial grid (superlattice) is formed and the example which has improved the property of a coat is also accepted. The present condition is that improvement is being made by these invention to the coat in which conventionally common TiN and a TiCN coat contain aluminum.

[0003]

[Problem(s) to be Solved by the Invention] however, the inclination for a cutting speed to become still quicker in the latest cutting in order to acquire high efficiency -- it is -- moreover, metal mold -- also in processing, although the soft steel before heat treatment was cut in many cases conventionally, the present condition is that the example which processes the high degree-of-hardness material after heat treatment directly is increasing in such high speed cutting and cutting of high degree-of-hardness material, although addition of aluminum raises the oxidation resistance of a coat and makes abrasion resistance improve rather than a TiN coat, it is now -- it is not enough satisfactory The coat in which the reason was generally formed of ion plating has compression residual stress, and the thickness of a coat follows this compression residual stress on becoming thick, and increases it. At most 5 micrometers of the thickness of the coat to which the adhesion deteriorates, therefore a coat can be equal to use in the present condition with the increase in compression residual stress are a limitation. For the reason, that abrasion resistance is inferior in the tool covered with ion plating compared with the covering tool which has the 10-15-micrometer thickness in which vacuum evaporation was carried out by the chemical vapor deposition (CVD) was the fact which cannot deny. Moreover, by formation of an artificial grid, it is a fact that the hardness of a coat improves, although wear-resistant improvement is accepted, such a stiff coat has high Young's modulus, and it has compression residual stress with a very high coat, and a limitation forms at most 3-5 micrometers. Since it has high compression residual stress, such an artificial grid coat has a big technical problem in adhesion.

[0004]

[Means for Solving the Problem] While this invention persons reduce residual compression stress, realize thick-film-ization in an ion plating coat and make abrasion resistance improve as a result Furthermore, by carrying out multilayer covering of two sorts of coats from which a stacking tendency differs, as a result of doing research which makes oxidation resistance improve Residual compression stress came to acquire the knowledge that oxidation resistance improves further, by being able to realize thick-film-ization and adding the 3rd predetermined \*\*\*\*\* to TiAl, without increasing.

[0005]

[Function] Generally in ion plating, the coat which a coat has a priority growth direction in a crystal growth, consequently has the pillar-shaped crystal structure is formed. If a crystal grain child pillar-shaped [ one ] is taken out, it is the single crystal with which a crystal growth strong against a fixed direction is accepted, and there are very few internal defects. That such a crystal forms membranes continuously is the cause which residual compression stress increases with the increase in the thickness of a coat. this invention person etc. came to develop the technology which introduces many lattice defects into the interface of a coat and a coat by carrying out multilayer covering of two sorts of coats from which a priority growth direction differs, respectively. That is, in carrying out multilayer covering of the nitride of Ti and aluminum which carry out orientation to the nitride, charcoal nitride, and field (200) of Ti which carries out orientation to a field (111), and the 3rd component, and the charcoal nitride, an interface becomes discontinuous, epitaxial growth is suppressed and many lattice defects are introduced. The rearrangement of the lattice defect of these many is carried out during growth so that the residual compression stress of a coat may be eased, and it suppresses the residual stress of a result and a coat and enables thick-film-ization. For example, when 0.5 micrometers of nitrides of Ti and aluminum which carry out orientation to (200), and the 3rd component are formed, residual stress is 1.2GPa, if 10 micrometers of this coat are formed, residual compression stress will exceed 8GPa and adhesion will deteriorate remarkably. When 0.5 micrometers of TiN(s) which carry out orientation of the nitride of Ti and aluminum which carry out orientation on the other hand (200), and the 3rd component to 0.5 micrometers and (111) are formed and a 10-micrometer coat is formed in multilayer

covering of this repeat, the residual compression stress is 2GPa(s) at most at a surprising thing. Therefore, according to this invention, thick-film-izing is easily possible, and it is possible to give very high abrasion resistance, without as a result degrading the adhesion of a coat in a covering tool.

[0006] Furthermore, it was admitted that the crack generated on the coat front face during cutting was in the inclination for the propagation to be suppressed, in the interface of the coat from which the direction of a crystal growth differs. That is, many lattice defects of an interface ease the stress concentration generated at the nose of a crack, and high resistance is shown to propagation of a crack. Simultaneously, a crack suppresses sharply the deficit of the edge of a blade which spreads in accordance with an interface and originates in the propagation to a base, and it, when progressing further. Therefore, since a crack cannot spread easily the multilayer covering hard tool which this invention twists while it has high abrasion resistance by thick-film-ization, it has high toughness simultaneously. Therefore, when a coat is thick and it is [ not only ] comparatively thin, raising a tool life cannot be overemphasized. Moreover, as a result of adding research to the oxidation-resistant improvement in the nitride of Ti and aluminum, and the charcoal nitride itself, it found out that oxidation resistance was improved remarkably by adding components, such as Zr, Hf, and Y.

[0007] Moreover, since residual compression stress is very small like the above-mentioned, the multilayer coat by this invention shows very high adhesion also to high-speed steel and a cermet alloy. Generally compressive stress generates the coat of ion plating during coating. Moreover, since high-speed steel and a cermet alloy have the coefficient of thermal expansion larger than a coat, in the cooling process after coating, compressive stress is further added to a coat. Consequently, when it takes out to a room temperature, it has compressive stress very higher than the case of a cemented carbide base, and a result and adhesion become remarkably bad. this invention multilayer coat also solves such a trouble.

[0008] Next, it is possible by making a coat contain carbon for a coat degree of hardness to improve remarkably and to make the further wear-resistant improvement realize. In this case, since the residual compression stress of the coat which contains carbon generally is remarkable and high when a carbon content is changed discontinuously, the interface intensity deteriorates remarkably. Therefore, the wear-resistant improvement by the improvement in hardness on the lubricous disposition of a coat is possible, without producing a problem in adhesion in making C<sub>2</sub>H<sub>2</sub> or a carbon source of supply called CH<sub>4</sub> increase gradually continuously, and making a coat contain carbon, in order to make carbon contain. Furthermore, the phenomenon in which wear advanced was found out by the repeat in which the porous oxide film which the coat oxidized and was formed during cutting is worn out. It is most effective to make 2Oaluminum3 most stable coat intervene to oxidization as a means to suppress oxidization of this coat further. This invention persons succeeded in forming 2Oaluminum3 extremely excellent coat of adhesion by forming aluminum 2O3 by plasma CVD or MOCVD, after forming the coat by ion plating. The wear by oxidization of a coat is sharply suppressed by formation of aluminum 2O3. The feed per revolution per one edge is comparatively high, and especially the coat especially by this invention has an effective base in the use like the insertion of cemented carbide or a cermet alloy, and a high-speed-steel end mill by which abrasion resistance and toughness (crack-proof propagation nature) are needed for a coat.

[0009] Hereafter, the reason which limited the numeric value is explained. In order to have compressive stress higher as this coat carries out orientation to a field (111) strongly, since it was [ the residual compression stress of this coat itself ] lower to carry out orientation to a field (200), the reason for having made the value of  $I(200)/I(111)$  of the nitride of Ti, aluminum, and the 3rd component and a charcoal nitride or more into one was preferably made or more into one that orientation should be carried out to a field (200). In order that the nitride of Ti and a charcoal nitride might introduce a lattice defect into an interface in the nitride of Ti and aluminum which carried out orientation to the field (200) as mentioned above, and the 3rd component, and multilayer covering with a charcoal nitride layer, orientation had to be made to carry out reversely to the nitride of Above Ti and aluminum, and a charcoal nitride in a field (111), and the value of  $I(200)/I(111)$  was made or less into one. If an effect will not be accepted at all to oxidation-resistant improvement if it is below 0.1 atom % in the amount of substitution, and 50 atom % is exceeded to Zr, Hf, Y, etc. of the 3rd component, in order to degrade the abrasion resistance which the nitride of Ti, or Ti and aluminum and a charcoal nitride originally have, it carried out to below 50 atom % more than 0.1 atom %. Hereafter, this invention is explained based on an example.

[0010]

[Example]

Example 1JIS By the arc ion plating method, Ti target and the TiAlMe target (Ti/aluminum=1, Me=5at%, Me=Zr, Hf, Cr, W, Y, Si) were used for the commercial high-speed-steel RAFINGU end mill of the cemented carbide insertion of P40 grade, and phi 12 or 4-sheet edge, and the coat shown in Table 1 was formed in it.

[0011]

[Table 1]

試料 番号	層厚 ( $\mu\text{m}$ )	層数	TiN	TiAlN	工具寿命 (m)		
			I(200) /I(111)	I(200) /I(111)	超硬インサート	HSS・EM	
本 発 明 例	1	8	16	0.5	2.5[Zr]	7.2	52.1
	2	8	16	0.5	3.2[W]	8.1	55.2
	3	8	16	0.1	1.8[Y]	9.5	53.2
	4	8	16	0.1	7.5[S1]	14.2	48.5
	5	13	26	0.5	8.2[Cr]	25.8	89.2
	6	5	10	0.5	7.5[Hf]	5.1	41.5
	7	3	8	0.5	6.3[Y]	2.2	30.2
	8	10	20	0.1	11.3[Zr]	9.5	68.2
	9	10	20	0.1	15.3[Y]	10.2	69.5
	10	10	40	0.1	15.3[Y]	13.5	71.2
比 較 例	11	3	1(TiN)	0.5	-	0.8	8.3
	12	10	1(TiN)	0.5	-	0.5 剥離	1.5 チッピング
	13	3	1(TiAl)N	-	10.2	1.5	21.2
	14	10	1(TiAl)N	-	10.2	0.5	10.2 チッピング

注意) [ ] : 添加成分を示す。添加量は5at%で一定。

[0012] TiN and the TiAlN coat were formed by the arc ion plating method same as a comparison tool. In the cemented carbide insertion, milling cutter cutting was performed based on cutting conditions 1, it asked with cutting length until a flank-wear value amounts to 0.3mm, and it was made into the life. Moreover, in the high-speed-steel end mill, it cut based on cutting conditions 2, and asked for the length of cut until a flank-wear value amounts to 0.2mm, and it was made into the life. The result is also written together to Table 1. Performing cutting conditions 1 by \*-ed material DAC(temper material) HRC= 40, cutting-speed 100 m/min and 0.1mm of delivery / edge, and 2mm of slitting, the configuration of an insertion used the SEE42-TN type. Cutting conditions 2 are \*-ed material DAC(green wood) HRC= 10, cutting-speed 50 m/min and 0.07mm of delivery / edge, 18mm of slitting shaft orientations, the 6mm of the directions of a path, cutting oil nothing, and Down. It carried out by Cut.

[0013] Also in thick-film-izing of 10 micrometers or more, neither exfoliation of a coat nor the chipping of the edge of a blade is accepted, but stable prolonged cutting is possible for the multilayer covering tool by this invention so that clearly from Table 1.

[0014] After forming the invention alloy shown in Table 2 using the arc ion plating method using the superhard insertion used in the example 2 example 1 like an example 1, 600 degrees C, 1hr, and alpha-aluminum 2O3 were formed as an outermost layer of drum on it by MO-CVD. By cutting conditions 1, the result which evaluated the life is also written together to Table 2.

[0015]

[Table 2]

試料	総膜厚 ( $\mu\text{m}$ )	層数	TiN I(200) /I(111)	TiAlN I(200) /I(111)	Al <sub>2</sub> O <sub>3</sub> 膜厚 ( $\mu\text{m}$ )	寿命 ( $\text{m}$ )	
本例 発明	15	10	20	0.5	9.2[Y]	0.5	21.5
	16	13	32	0.5	4.1[Y]	0.5	38.2
	17	5	20	0.1	15.2[Y]	0.5	15.1
比較	18	10	1(TiAl)N	-	11.5	-	1.2剥離発生
例	19	3	1(TiAl)N	-	8.8	-	1.2

注意) [ ] : 添加成分を示す。添加量は5at%で一定。

[0016] When this invention tool forms 2Oaluminum3 coat, reinforcement is further attained, so that clearly from Table 2.

[0017] In this invention alloy of the sample number 1 made as an experiment in the example 3 example 1, the carbon content was made to increase [ be / under / coat / continuation / it ], and the tool which made carbon contain was made as an experiment. The contents are shown in Table 3.

[0018]

[Table 3]

試料		層厚 ( $\mu\text{m}$ )	層数	a	b	TiN I(200) /I(111)	TiAlN I(200) /I(111)	最終膜 C/N	工具寿命 (m)	
									超硬インサート	HSS・EM
本 発 明 例	1	8	16	-	-	0.5	1.9[Zr]	-	6.2	51.2
	20	8	16	0.5	5.8	0.6	2.1[Zr]	1/1	8.8	53.6
	21	8	16	0.5	7.0	0.6	2.1[Y]	1/1	14.2	60.1
	22	8	16	3.2	7.0	0.6	2.1[W]	1/2	7.1	55.1
比 較	23	8	1(TiN)	-	-	-	1.8	-	0.2 剥離	1.2 チッピング
	24	5	1(TiN)	-	-	-	6.1	-	1.7	22.2

注意) [ ] : 添加成分を示す。添加量は5at%で一定。

[0019] Among Table 3, the thickness of the coat at the time of alpha beginning to add C2H2 and b increase C2H2 amount gradually, and show the thickness of the coat at the time of supposing that it is fixed after that. That is, between a-b is the layer which a carbon content increases continuously. In this case, thickness of each class of the nitride of Ti or the charcoal nitrides Ti

and aluminum and the nitride of the 3rd component, or a charcoal nitride was set to 0.5 micrometers, respectively. In the cutting conditions 1 and 2 used in the example 1, the cutting test was performed to the superhard insertion and high-speed-steel end mill, and the life was searched for. The result is also written together to Table 3.

[0020] Further wear-resistant improvement was checked by this invention alloy by content bundle \*\*\*\*\* in carbon so that clearly from Table 3.

[0021] The target of the 3rd component was installed for one of the best stage using the arc ion plating system which has the target of 46 examples (three step x2 train), and invention of the composition which uses three of Ti target and an opposite train as a TiAl target, and shows two under it in Table 4 was made as an experiment.

[0022]

[Table 4]

試料	組成 - 1	組成 - 2	被膜厚 ( $\mu\text{m}$ )	層数	TiN I(200) /I(111)	TiAlN I(200) /I(111)	酸化 膜厚 ( $\mu\text{m}$ )	寿命 (m)
本 発 明 例	1 (Ti <sub>0.8</sub> Al <sub>0.2</sub> )N	TiN	8	16	0.5	1.5	1.4	5.7
	25 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Zr <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.2	6.3
	26 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Hf <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.5	7.2
	27 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Cr <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.4	6.2
	28 (Ti <sub>0.4</sub> Al <sub>0.4</sub> W <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.3	7.8
	29 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Y <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.1	10.3
	30 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Si <sub>0.2</sub> )N	"	8	16	0.6	1.7	0.2	11.2
	31 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Co <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.1	8.2
	32 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Nd <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.1	7.8
	33 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Zr <sub>0.2</sub> )N	"	8	16	0.2	3.5	0.15	5.0
	34 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Y <sub>0.2</sub> )N	"	8	16	0.2	4.2	0.05	4.8
	35 (Ti <sub>0.4</sub> Al <sub>0.4</sub> Y <sub>0.2</sub> )N	"	8	16	0.5	1.5	0.15	8.0
	36 TiN	-	8	1	0.5	-	7.5	0.8
	37 (Ti <sub>0.8</sub> Al <sub>0.2</sub> )N	-	8	1	-	1.5	1.3	0.5 剥離
	38 (Ti <sub>0.8</sub> Al <sub>0.2</sub> )N	-	8	1	-	2.0	1.5	1.7

[0023] The 3rd component ratio was suppressed by adjusting arc current Ti simultaneously passed at the 3rd component target, although it changed with the distance from the 3rd component target. It was made to oxidize by having carried out 1hr maintenance of these examples of this invention, and the example of comparison at 750 degrees C among the atmosphere, and oxidation thickness was suppressed. The result is written together to Table 4. Moreover, the result which carried out life evaluation in the insertion is also written together by cutting conditions 1.

[0024] From Table 4, oxidation-resistant remarkable improvement is accepted by addition of the 3rd component, and improvement in the life which originates in oxidation-resistant improvement simultaneously is checked.

[0025] example 525TiC-30TiN-20WC-5TaC-5Mo2C-8Co-7nickel (% of the weight) -- a prototype was built from the contents which show the cermet alloy of composition in Table 5, and it coated like the example 1

[0026]

[Table 5]

試料	被膜厚 ( $\mu\text{m}$ )	層数	TiN I(200) /I(111)	TiAlN I(200) /I(111)	工具寿命 (m) サーメット インサート
本 発 明 例	36 8	16	0.5	2.5[Zr]	18.1
	37 8	16	0.5	3.2[W]	21.2
	38 8	16	0.1	1.8[V]	29.5
	39 8	16	0.1	7.5[Si]	35.1
	40 13	26	0.5	8.2[Cr]	42.1
	39 3	1(TiN)	0.5	-	8.2
比 較 例	40 10	1(TiN)	0.5	-	0.1 剥離
	41 3	1(TiAl)N	-	10.2	10.8
	42 10	1(TiAl)N	-	10.2	0.1 剥離

注意) [ ] : 添加成分を示す。添加量は 5 at % で一定。

[0027] In the insertion, cutting conditions 3 performed cutting evaluation and it considered as the life in quest of the length of cut until the amount of flank wears amounts to 0.15mm. The result is also written together to Table 5. An insertion (SEE42-TN) is used for cutting conditions 3, and they are \*\* material SKD 61 (green-wood HRC12)-ed, cutting-speed 250 m/min, and a feed per revolution. 0.1mm / edge, the amount of slitting It is 2mm. The size of \*\*-ed material is the same as that of an example 1. In this invention multilayer covering cermet, there is no exfoliation of a coat during cutting and long lasting achievement is possible as shown in Table 5.

[0028]

[Effect of the Invention] While easing the residual compression stress accompanying the increase in the thickness of a coat by applying this invention like the above, thick-film-ization was completed, and further, since it was hard to spread a crack, without

degrading membranous adhesion, the coat which has high toughness simultaneously was obtained. Moreover, a coat degree of hardness, lubricity, and oxidation resistance can be notably raised by combining carbon with the 3rd element, an oxide coat, and a coat for content etc.

---

[Translation done.]

**\* NOTICES \***

**Japan Patent Office is not responsible for any damages caused by the use of this translation.**

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

**CLAIMS**

---

[Claim(s)]

[Claim 1] In the multilayer covering hard tool which covered at least the layer which consists of a Ti nitride or a charcoal nitride, and the layer which consists of the nitride or charcoal nitride of Ti, aluminum, and a third component more than two-layer When intensity of I (111) and a field (200) is set to I (200) for the intensity of the field in an X diffraction (111), The nitride of Ti or the value of I (200)/I (111) of a charcoal nitride layer is one or less. It is the multilayer covering hard tool by which the nitride of Ti, aluminum, and a third component or the value of I (200)/I (111) of a charcoal nitride layer is one or more, and a third component is characterized [ which are one sort or two sorts or more of components chosen from Zr, Hf, Cr, W, Y, Si, Ce, and Nb ].

[Claim 2] The multilayer covering hard tool characterized by the content of carbon having the layer which increases from a base front face continuously in the direction of a coat front face in a multilayer hard tool according to claim 1.

[Claim 3] The multilayer covering hard tool by which one sort of Zr, Hf, Cr, W, Y, Si, Ce, and Nd or two sorts or more of additions are characterized by being the range of 0.1 atomic ratios to 50 atomic ratios to Ti and aluminum in a multilayer covering hard tool according to claim 1 to 2.

[Claim 4] The multilayer covering hard tool characterized by having at least one layer of oxide layers of aluminum in a multilayer covering hard tool according to claim 1 to 3.

[Claim 5] The multilayer covering hard tool which has that a base is a cemented carbide insertion in a multilayer covering hard tool according to claim 1 to 4.

[Claim 6] The multilayer covering hard tool characterized by a base being a high-speed-steel end mill in a multilayer covering hard tool according to claim 1 to 4.

[Claim 7] The multilayer covering hard tool characterized by a base being a cermet insertion in a multilayer covering hard tool according to claim 1 to 4.

---

[Translation done.]